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IS 1448-83 (1974): Methods of test for petroleum and its products, Part 83: Determination sulphur by Wickbold oxyhydrogen method [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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IS : 1448 [P : 83] - 1974
Adopted : 30 *December* 1974

(Reaffirmed 2002)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS : DETERMINATION OF SULPHUR BY WICKBOLD OXY-HYDROGEN METHOD

[P : 83]

(Second Reprint APRIL 1986)

UDC 665·6/·7 : 543·845

Adapted from IP 243/70T Sulphur in Petroleum Products Wickbold Oxy-Hydrogen Method.

1. SCOPE

1.1 This method describes procedures for the determination of total sulphur in petroleum products. These procedures are applicable to volatile sulphur in natural, refinery, and liquefied petroleum gases; and to total sulphur in light distillate feed-stocks, solvents, gasolines, kerosines, gas oils, fuel oils, crudes, residues, and some lubricating oils. The method is particularly suitable for distillates with total sulphur contents of less than 300 ppm. Samples which are viscous, aromatic, or of high sulphur content are first diluted with a solvent.

1.2 In the thorin finish, chlorine and nitrogen in the sample may exceed the sulphur concentration by several times without causing interference. Fluorine interferes, if present in concentrations higher than 30 percent of the sulphur content. Phosphorus and cations interfere. Sodium, potassium, zinc, magnesium, and ammonium ions reduce results by 2 to 3 percent due to co-precipitation. Cations forming insoluble sulphates interfere quantitatively and many other metal ions interfere by forming coloured complexes with thorin in alcoholic solutions. However, in the amounts normally present in products these interferences are not significant but do preclude the use of the method on most lubricating oils containing additives.

2. OUTLINE OF THE METHOD

2.1 The sample, diluted if necessary with a suitable solvent, is burned in a rapid combustion burner. The products of combustion are drawn through

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Gr 4 *May* 1975

dilute hydrogen peroxide solution which is analysed for sulphate. For samples having sulphur contents in the range 0 to 10 ppm the sulphate is determined by nephelometric titration. For higher concentrations the quicker barium perchlorate/thorin titration is used.

3. APPARATUS

3.1 An assembly of the Wickbold combustion apparatus as shown in Fig. 1 shall include the components described below.

3.1.1 *Stand with safety Shield* — The safety shield shall be made of colourless safety glass or its equivalent and mounted in front of and at the sides of the apparatus.

3.1.2 *Burner* — Either quartz Wickbold burner or a Fisher stainless steel burner provided with inlets for primary oxygen, secondary oxygen, hydrogen and the sample. (These burners will normally burn 5 to 6 ml of iso-octane per minute.) Flow diagrams are given in Fig. 2A and 2B.

3.1.3 *Combustion Tube with Condenser* — made of quartz.

3.1.4 *Flowmeter and Metal Control Valve* (1 000 *l/h*) — of safe design for primary oxygen, and capable of measuring 1 000 *l/h*. A suitably calibrated mercury manometer is satisfactory.

3.1.5 *Flowmeter and Metal Control Valve* (100 *to* 400 *l/h*) — of safe design for secondary oxygen, and capable of measuring 100 to 400 l/h. A suitably calibrated mercury differential manometer is satisfactory.

3.1.6 *Flowmeter and Metal Control Valve* (50 *to* 100 *l/h*) — of safe design capable of measuring 50 to 100 l/h of hydrogen.

3.1.7 *Mercury Manometer* — 400 mm in height, filled to a height of 200 mm and provided with a millimetre scale.

3.1.8 *Vacuum Needle Control Valves*

3.1.9 *Flask* — Three-necked, 500 ml capacity, fitted with a stopcock.

3.1.10 *Absorption Tower* — provided with sintered filter plate (porosity 1) and cooling jacket.

3.1.11 *Reitmeyer Spray Trap*

3.1.12 *Metal Pressure Safety Valves* — for use in the primary and secondary oxygen lines and in the hydrogen line, 0·92 bar (0·9 atm) excess pressure.

3.1.13 *Flow Indicator* — glass.

3.2 Vacuum Pump — capable of drawing at least 1 500 l/h through the apparatus, protected by a water trap.

3.3 Oxygen Cylinder Reducing Head

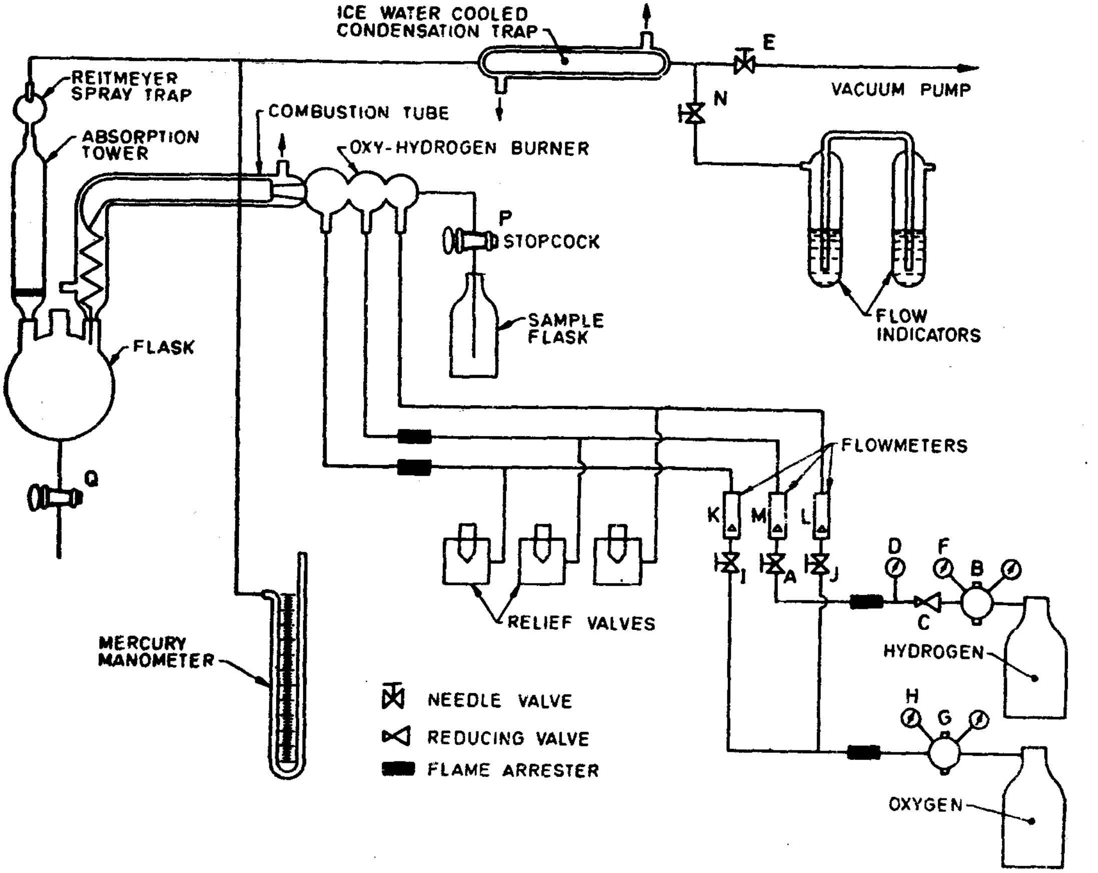

FIG. 1 WICKBOLD RAPID COMBUSTION APPARATUS FOR LIQUID SAMPLES OTHER THAN LPG

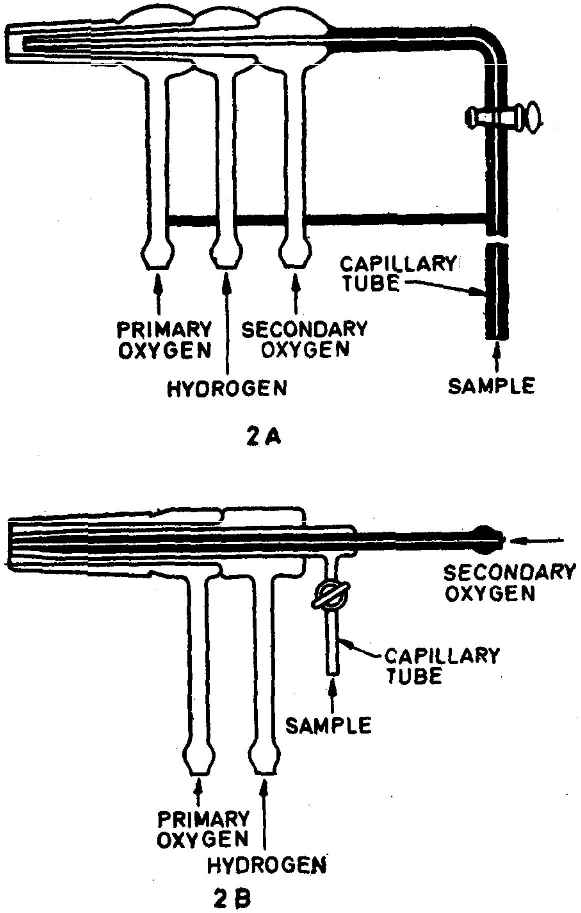

FIG. 2 ALTERNATE TYPES OF QUARTZ BURNER

3.4 Hydrogen Cylinder Reducing Head

3.5 Reducing Valve — for further reduction of hydrogen pressure from 3·45 to 0·345 bar.

3.6 Pressure Gauge — for measuring hydrogen pressure after final reduction.

3.7 Daylight Blue Lamp — 100 watt.

3.8 Flame Arresters — of sheet, fine mesh copper gauze, 650 mm^2 in area rolled to fit into the hydrogen supply line in the positions indicated in Fig. 1.

3.9 Precision Needle Valve — for controlling flow of test gas (Fig. 3 and Fig. 4). This is to be constructed from materials which will not react with sulphur compounds in the gas.

3.10 Nylon or Silicone Rubber Tubing — suitable for housing the needle valve (Fig. 4).

3.11 Water-Bath — suitable for housing the needle valve (Fig. 4).

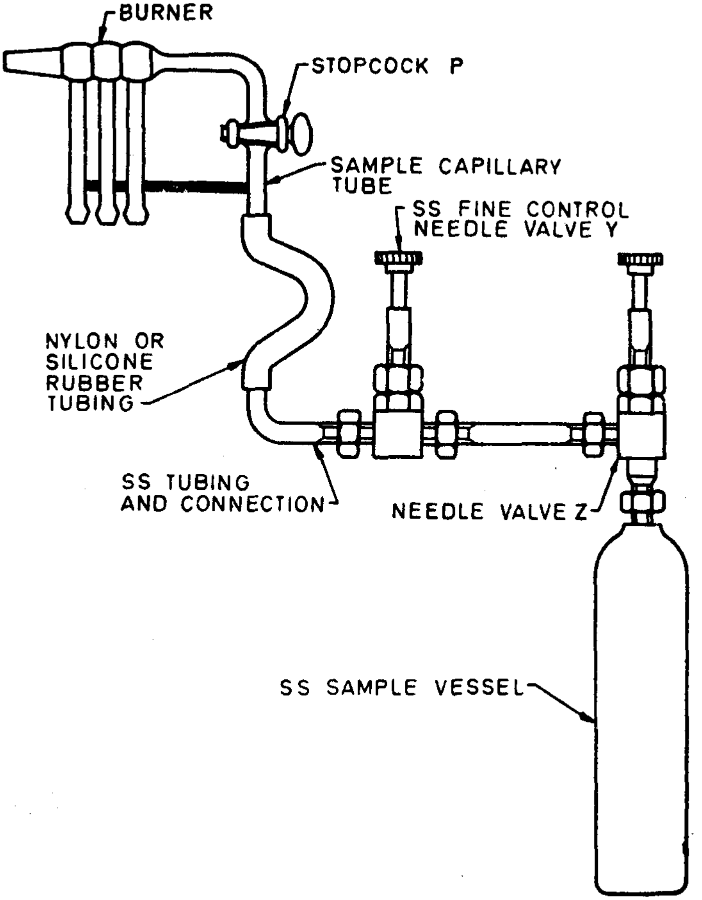

FIG. 3 GAS FEED TO BURNER

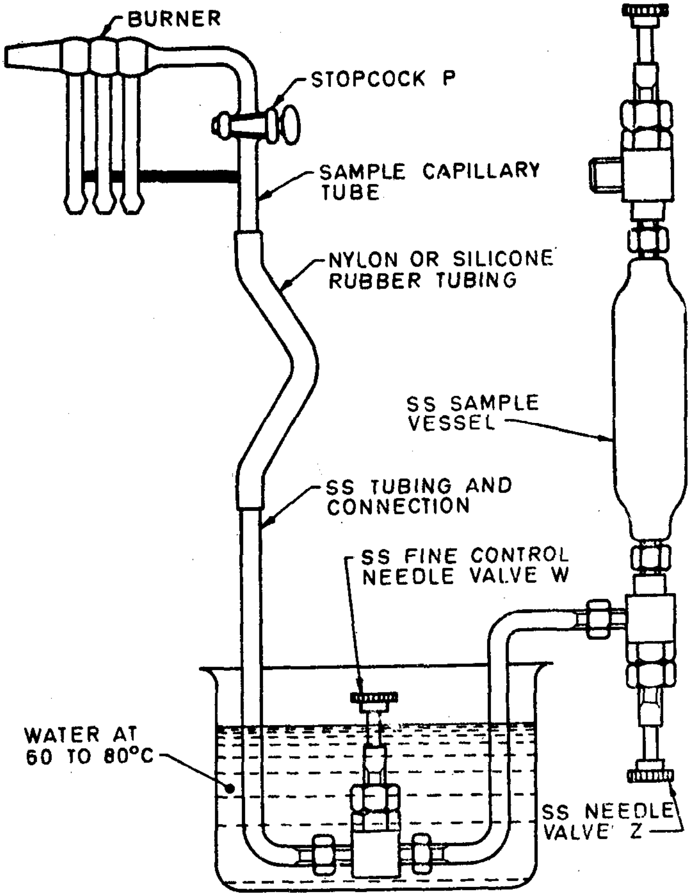

FIG. 4 LIQUEFIED GAS FEED TO BURNER

3.12 Nephelometer and Galvanemeter — alternatively a photoelectric colorimeter which can be suitably adapted to the technique described.

3.13 Erlenmeyer Flask — 250 ml with B24 ground-glass socket and fitted with a B24/29 standard quick-fit cone joint and of total length 150 mm to act as a chimney.

4. REAGENTS AND MATERIALS

4.0 Quality of Reagents — Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

4.1 For Combustion Procedure

4.1.1 *Oxygen* — compressed.

4.1.2 *Hydrogen* — compressed.

4.1.3 *Hydrogen Peroxide* — 1·5 percent (m/v).

4.1.4 *Iso-octane* — Knock test grade or petroleum hydrocarbon solvent 60/80 conforming to IS : 1745-1966†, with a sulphur content of less than 1 ppm as determined by this method.

4.1.5 *Toluene* — Sulphur content less than 1 ppm as determined by this method.

4.1.6 *Diluent* — mixture of equal volumes of iso-octane (**4.1.4**) and toluene (**4.1.5**).

4.2 For Thorin Titration

4.2.1 *Acetone* — *see* IS : 170-1966‡.

4.2.2 *Isopropyl Alcohol* — *see* IS : 2631-1964§.

4.2.3 *Thorin Indicator Solution* — 0·2 percent (m/v) aqueous solution of thorin [1-(o-arsenophenyl-azo)-2- naphthol-3 : 6-disulphonic acid sodium salt]. Store away from light and label ' CAUTION — POISON '.

4.2.4 *Standard Sulphuric Acid Solution* — 0·005 N.

4.2.5 *Standard Barium Perchlorate Solutions* — 0·005 N and 0·01 N. Dissolve 0·975 and 1·95 g respectively of barium perchlorate trihydrate in 200 ml of distilled water and make up to 1 litre with isopropyl alcohol. Adjust the apparent *p*H of the solution to between 3 and 4 with a few drops of 10 percent perchloric acid.

CAUTION — Barium perchlorate should not be heated in the presence of organic substances, or brought into contact with acids as it may explode.

4.2.5.1 To standardize, pipette 5 ml of the standard 0·005 N sulphuric acid solution into the 500-ml flask. Pipette in 25 ml of distilled water and

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*Specification for water, distilled quality (*revised*).

†Specification for petroleum hydrocarbon solvents (*first revision*).

‡Specification for acetone (*first revision*).

§Specification for isopropyl alcohol.

add, from a measuring cylinder, 120 ml of acetone. Add 1 or 2 drops of thorin indicator, swirl to mix. Titrate this solution with barium perchlorate until the solution turns from yellow to pink (*see* Note). Calculate the normality of the barium perchlorate solution from the mean of 5 such titrations. Re-standardize every 10 days.

NOTE — The thorin end-point is very difficult to see in some types of light, particularly fluorescent lighting and in direct sunlight. Good results can be obtained by titrating in front of a daylight blue lamp.

If an indefinite colour change is obtained, the indicator should be considered suspect and a fresh solution prepared. In some cases the end-point can be improved by the addition of five drops of 0·01 percent aqueous methylene blue solution.

4.3 For Nephelometric Titration

4.3.1 *Methanol — see* IS : 517-1967*.

4.3.2 *Sodium Chloride Solution* — 10 g/l.

4.3.3 *Standard Sulphuric Acid Solution* — 0·005 N.

4.3.4 *Standard Barium Chloride Solution* — 0·005 N. Dissolve 0·521 g of anhydrous barium chloride in distilled water and make up to 1 litre. To standardize, pipette 2·00 ml of the standard 0·005 N sulphuric acid solution into a 250-ml Erlenmeyer flask containing about 20 ml of distilled water. Add 1·0 ml of the sodium chloride solution, place one of the special chimneys in the neck of the flask and proceed with the evaporation step and subsequent titration procedure as detailed in **7.11.1**.

4.3.4.1 Carry out a blank determination omitting the 2·00 ml of standard sulphuric acid solution. Calculate the normality of the barium chloride solution.

4.3.5 *Coagulant Solution* — Dissolve 200 g of magnesium chloride hexahydrate and 35 g of ammonium chloride in 1 litre of distilled water. Dilute to one-tenth of the concentration when required for use and adjust *p*H to between 8 and 8.2 with ammonium hydroxide.

5. SAMPLE

5.1 Obtain samples of liquid and natural, refinery or other gaseous fuels including LPG in previously cleaned containers by the appropriate procedures described in IS : 1447-1966†.

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*Specification for methanol (methyl alcohol) (*first revision*).

†Methods of sampling of petroleum and its products.

6. PREPARATION OF APPARATUS

6.1 Scrupulous cleanliness of sample containers and apparatus is required throughout. The sample containers, lines, valves, and connectors used for gas and LPG samples should be washed with sulphur-free toluene until the sulphur content of the washings as determined according to the procedure for liquid samples is negligible. Ensure that the containers are solvent-free before taking samples. Glassware and quartzware may be cleaned with concentrated detergent but shall be finally thoroughly washed with water to remove sulphur compounds. More rigorous cleaning may be accomplished with dichromate/nitric acid cleaning agent. Quartzware may be cleaned by heating in a muffle furnace.

6.1.1 It is preferable to reserve a set of sample containers and quartz and glassware equipment for low sulphur content samples and keep another set for high sulphur content samples.

6.2 Assemble the apparatus as shown in Fig. 1 and the gas supplies as in Fig. 2A or Fig. 2B. Use high pressure copper tubing to connect the oxygen and hydrogen supply to the control valves *A*, *I*, and *J*. Fill both limbs of the flow indicator with water to a height of approximately 30 mm. Suspend the burner from a piece of wire of such length as to permit easy manipulation.

CAUTION — Ensure that the safety shield is in position.

6.3 Close the oxygen and hydrogen control valves *A*, *I* and *J*. Adjust the reducing head valve *B* of the hydrogen cylinder until the gauge *F* shows a pressure of 3·45 bar. Now adjust the hydrogen control valve *C* until the pressure gauge *D* shows a pressure of 0·345 bar. Adjust the oxygen reducing head valve *G* until the gauge *H* shows a pressure of 0·69 bar. Pass a rapid stream of water through the condenser and absorption tower and iced water through the condensation trap. Close valve *E* and switch on the vacuum pump.

7. PROCEDURE

7.1 Select the appropriate sample size from Table 1 for distillate fuels with sulphur contents of 300 ppm or less and from Table 2 for other samples. For liquid samples proceed to **7.2**. For gaseous fuels and liquefied petroleum gases proceed directly to **7.3**.

7.2 For Liquid Samples Only — Prepare the sample as follows. For distillates, weigh the sample directly into a conical flask. For other samples, including lubricating oils, residual fuels, and crudes, measure 20 ml of the diluent into a conical flask and weigh in the appropriate amount of sample. Quickly cover the mouth of the conical flask with aluminium foil.

CAUTION — To avoid any risk of explosion, very volatile liquids, such as iso-pentane, shall be diluted with an equal volume of sulphur-free iso-octane (*see* Note *under* **7.4**). Smoother combustion may be obtained when testing gas oils or highly aromatic samples by a similar dilution.

TABLE 1 SAMPLE SIZE FOR LOW SULPHUR CONTENTS

(*Clause* 7.1)

TOTAL SULPHUR CONTENT	MASS OF SAMPLE (TITRATION METHOD)		NORMALITY OF BARIUM PERCHLORATE FOR THORIN TITRATION
	Nephelometric	Thorin	
(1)	(2)	(3)	(4)
ppm by mass	g	g	
Below 10	100 to 40	—	—
10 to 30	—	40 to 20	0·005
30 to 100	—	30 to 10	0·01
100 to 300	—	10 to 5	0·01

TABLE 2 SAMPLE SIZE FOR HIGH SULPHUR CONTENTS

(*Clause* 7.1)

TOTAL SULPHUR CONTENT	MASS OF SAMPLE
(1)	(2)
percent by mass	g
0·03 to 0·2	5 to 1
0·2 to 0·5	1 to 0·3
0·5 to 1·0	0·3 to 0·15
1·0 to 5·0	0·15 to 0·05

7.3 For All Samples — Insert the burner, close the burner stopcock *P* and open the flow indicator stopcock *N*.

CAUTION — It is imperative to take extreme care and to wear safety goggles during the combustion stage.

7.4 Pass primary oxygen through the apparatus at a rate of 16 l/min and secondary oxygen at a rate of approximately 3 l/min (*see* Note), at the same time opening the vacuum control valve *E* until flow through the flow indicator stops. Then close stopcock *N* and adjust the vacuum control valve *E* to obtain a pressure of 26·7 ± 6·7 mbar (20 ± 5 mmHg) below atmospheric in the three necked flask as indicated by the manometer.

NOTE — The flow rates are given for guidance and may have to be adjusted slightly to give the best results for very volatile liquids such as iso-pentane. To improve combustion with highly aromatic samples increase the secondary oxygen rate slightly and adjust the vacuum accordingly.

7.5 Remove the spray trap from the absorber column and hold in one hand; with the other hand pour 30 ml of the hydrogen peroxide solution into the absorber column. Replace the spray trap.

7.6 Take out the burner. Open the hydrogen valve *A* until a hydrogen flow rate of 0·8 l/min is achieved. Allow the hydrogen to pass for 30 seconds. Now ignite the hydrogen with a flint or battery operated lighter or wax taper (do not use matches). Place the burner in the combustion tube taking care that the flame does not touch the wall of the standard joint. The pressure as measured by the manometer will not drop. Restore the pressure to approximately 26·7 mbar (20 mmHg) below atmospheric by manipulating the vacuum control valve *E*.

7.7 According to the type of sample, proceed as directed in **7.7.1** for gaseous fuels, in **7.7.2** for liquefied petroleum gases, and in **7.7.3** for liquid samples.

7.7.1 *For Natural and Refinery Gases* — Proceed as follows.

7.7.1.1 Weigh the sample container to the nearest 0·1 g and connect the sample container to the capillary tube of the burner as shown in Fig. 3. Open stopcock *P* fully. Start combustion by opening valve *Z* (Fig. 3) fully, followed by valve *Y* slowly until the flame length is approximately three-quarters of the length of the combustion tube. Start the stopclock. Further adjustments may be made using the vacuum control valve *E*.

CAUTION—Should the burner flame extinguish at any time, first turn off the hydrogen at valve *A*, turn off the sample supply at source, remove the burner and then quickly turn off the oxygen at valve *G*. Abandon the determination and prepare the apparatus for another determination as described in **6**.

7.7.1.2 When sufficient gas has been burnt, shut off valve *Z* and open valve *Y* (Fig. 3). Close stopcock *P* and disconnect the sample line. Continue as given in **7.8**.

7.7.2 *For Liquefied Petroleum Gases* — Proceed as follows.

7.7.2.1 Weigh the sample container and contents to the nearest 0·1 g and connect the sample container to the capillary tube of the burner as shown in Fig. 4.

7.7.2.2 Fill the water-bath with water at 80°C and maintain the temperature between 60 and 80°C during combustion.

7.7.2.3 Start the combustion by fully opening stopcock *P* and valve *Z*, followed by valve *W* slowly, until the flame length is approximately three-quarters of the length of the combustion tube (*see* Note *under* **7.7.1.1**). Start the stopclock. Further minor adjustments may be made using the vacuum control valve *E*.

7.7.2.4 When sufficient sample has been burnt, shut valve *Z*; disconnect the sample container and allow the remaining vapours in the connecting manifold to be drawn through the burner. Shut stopcock *P* and disconnect the sample line. Continue as given in **7.8**.

7.7.3 *For Liquid Samples* — Proceed as follows.

7.7.3.1 Pierce the aluminium foil covering the neck of the sample container and place the container in position with the capillary tube reaching to the bottom of the sample.

7.7.3.2 Start the combustion by partially opening the burner stopcock *P*. Adjust the flame length using the burner stopcock *P*, until the flame length is approximately three-quarters of the length of the combustion tube (*see* Note *under* **7.7.1.1**). Start the stopclock. If the burner stopcock *P* is fully open further adjustment may be made using the vacuum cotrol valve *E*.

7.7.3.3 When the sample has been burnt, wash the sample bottle with three successive 2-ml portions of iso-octane or diluent solvent as appropriate. Allow each portion to burn, keeping the sample inlet filled all the time. Remove the sample bottle and gradually open the burner stopcock until the burner is emptied by suction taking care to maintain a slight vacuum in the three-necked flask. Close the burner stopcock *P*.

7.8 Restore the pressure in the three-necked flask to approximately 26·7 mbar (20 mmHg) below atmospheric and close the hydrogen valve *A*. Allow the burner to remain in place until the flame is extinguished. Stop the stopclock. Take the burner out of the combustion tube, close the oxygen valves, *I* and *J* and the vacuum control valve *E*. Open the flow control indicator stopcock *N*. Switch off the vacuum pump. Lift the spray trap and allow the absorber solution to drain into the receiving flask. Remove the adaptor from the central neck of the flask.

7.9 For Natural, Refinery, and Liquefied Petroleum Gases only — Reweigh the sample container.

7.10 Blank Determinations

7.10.1 Irrespective of finishing procedure make a blank determination by running the apparatus for an appropriate time with the oxy-hydrogen flame alight and including in the combustion the same amount of diluent as used during the sample combustion procedure.

7.10.2 Prepare the products of combustion for the required titration procedure as laid down in **7.11.1** or **7.11.2** respectively and complete the titration.

7.10.3 Redetermine the blank whenever new reagents are used.

7.11 Proceed to the finish required.

7.11.1 *Nephelometric Titration Finish* — Transfer the contents of the three-necked flask to the Erlenmeyer flask by drain stopcock *Q*.

7.11.1.1 Wash down the splash trap, absorber tower and combustion/condenser tube with two 25-ml portions of distilled water. Collect the washings in the three-necked flask, swirl, and transfer to the Erlenmeyer flask.

Wash the three-necked flask with 25 ml of distilled water and add the washings to the Erlenmeyer flask. Add 1 ml of the sodium chloride solution. Place the special chimney in the neck of the Erlenmeyer flask. Evaporate the solution just to dryness on a hotplate. Transfer to an electric oven at 150°C for a minimum of 20 minutes to ensure final removal of hydrochloric and nitric acids.

7.11.1.2 Take the flask from the oven, allow to cool, remove the chimney and add 1 ml of the coagulant solution and 3 ml of methanol to dissolve the residue. Transfer the liquid to the test tube of the nephelometer. Rinse the Erlenmeyer flask twice with a further 7 ml of methanol in two successive portions and add the washings to the test tube. Stir gently with a glass rod, remove the rod to a clean beaker, and measure the initial turbidity of the solution.

7.11.1.3 Titrate with the standard 0·005 N barium chloride solution in small increments (0·1 ml) from a microburette. Stir gently with the glass rod between each addition and record the turbidity and burette readings. Continue the titration until successive turbidity readings become constant or show a steady fall with equal additions of barium chloride. Plot a graph from the readings and determine the precise end point; this is the intersection of two straightlines joining the plots before and after the end-points.

7.11.2 *Thorin Titration Finish* — Transfer the liquid in the three-necked flask to a conical flask through stopcock *Q* and evaporate to a convenient volume but not less than 30 ml. Rinse down the spray trap, absorber column and combustion tube with a volume of acetone equal to approximately 4 times the volume and allow the washings to drain into the three-necked flask. Transfer the washings to the conical flask (*see* Note). Add 1 or 2 drops of the thorin indicator to the flask, swirl to mix, and titrate with the appropriate strength of barium perchlorate solution (Table 1) until the yellow colour shows the first trace of pink, using a similar volume of yellow solution for guidance. It is preferred that a magnetic stirrer is used to stir the contents of the flask during titration.

NOTE — At this stage the *p*H of the solution is between 2·0 and 5·0 using the indicator paper. Adjust *p*H if necessary with perchloric acid.

8. CALCULATION

8.1 Nephelometric Finish

$$\text{Total sulphur content, ppm} = \frac{(A - B) \times N \times 1{\cdot}6 \times 10^4}{M}$$

where

A = volume in ml of standard barium chloride solution used in the determination;

B = volume in ml of standard barium chloride solution used in the blank;

N = normality of the standard barium chloride solution; and

M = mass in g of sample used in the determination.

8.2 Barium Perchlorate/Thorin Finish

$$\text{Total sulphur content, ppm} = \frac{(A - B) \times N \times 1{\cdot}6 \times 10^4}{M}$$

or

$$\text{Total sulphur content, percent by mass} = \frac{(A - B) \times N \times 1{\cdot}6}{M}$$

where

A = volume in ml of standard barium perchlorate used in the determination;

B = volume in ml of standard barium perchlorate used in the blank;

N = normality of the standard barium perchlorate solution; and

M = mass in g of sample used in the determination.

9. REPORTING

9.1 Report the result as volatile sulphur for natural, refinery, and liquefied petroleum gases and as total sulphur for liquid samples parts per million by mass or percent by mass.

10. PRECISION

10.1 Results of duplicate tests shall not differ by more than the following amounts:

a) *Nephelometric Finish*

Range of Evaluation	*Repeatability*	*Reproducibility*
0·4 to 9·0 ppm	0·17 x ppm	1·7 ppm

b) *Thorin Finish* (Range of Evaluation 7 ppm to 4·0 percent by mass)

Levels, percent by mass x	*Repeatability* $0{\cdot}074\,x^{0{\cdot}85}$	*Reproducibility* $0{\cdot}24\,x^{0{\cdot}86}$
0·001	0·0002	0·0007
0·01	0·0015	0·0048
0·1	0·011	0·033
0·5	0·041	0·13
1·0	0·074	0·24
2·0	0·13	0·43
3·0	0·19	0·59
4·0	0·24	0·78

where

x represents the mean.

NOTE — The precision values have been obtained by statistical examination of inter-laboratory test results. Gases and liquefied petroleum gases were not included in this programme but it is expected that the precision on these types of sample would be equivalent to or better than the precision quoted.

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